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AD828473

# JET FUEL STABILITY AND EFFECT OF FUEL-SYSTEM MATERIALS

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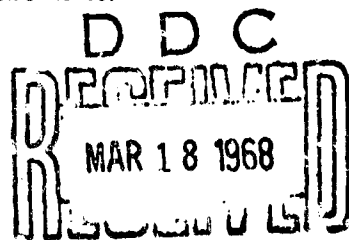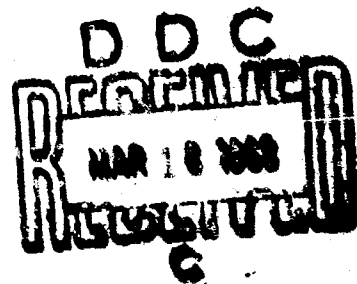
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# JET FUEL STABILITY AND EFFECT OF
# FUEL-SYSTEM MATERIALS

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# NOTICES

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# FOREWORD

This report was prepared by Southwest Research Institute, San Antonio, Texas, under Contract AF 33(615)-2327. The contract was initiated under Project Nos. 8169 and 3048, Task Nos. 816901 and 304801. The work was performed by contractor's personnel using Air Force facilities at Wright-Patterson AFB and was administered by the Ground Support Branch and the Fuels, Lubrication, and Hazards Branch, Support Technology Division, Air Force Aero Propulsion Laboratory, Air Force Systems Command, Wright-Patterson AFB, Ohio. The project engineers were Mr. John L. Morris and Mr. Paul C. Linder (APFL).

This report covers one phase of the work performed under the subject contract between 1 March 1966 and 30 November 1967. This report was submitted by the authors on 31 January 1968. Contractor's identifying numbers for this report are Project No. 09-1663, Report No. RS-515.

The authors acknowledge with thanks the advice and guidance provided by the project engineers and other Air Force personnel. The authors also acknowledge with thanks the contribution of Dr. W. G. Scribner of Monsanto Research Corporation in the performance and interpretation of the analyses for metal contents.

This technical report has been reviewed and is approved.


Arthur V. Churchill, Chief
Fuels, Lubrication, and Hazards Branch
Support Technology Division
Air Force Aero Propulsion Laboratory

ii

# ABSTRACT

A thermally stable, low volatility, naphthenic type jet fuel was stored in lined steel drums for one year at 130°F with various metallic and non-metallic specimens representing ground fuel system materials. Fuel thermal stability was measured periodically by means of a gas-drive fuel coker. The fuel itself remained thermally stable during storage and was not degraded seriously by steels, aluminums, coated steels, plug valve grease, or a filter-separator element. Water rusty steel, when present during storage, degraded the fuel thermal stability significantly. Brass, bronze, and butadiene-acrylonitrile rubbers of the type used for fuel hose gave the worst degradation of fuel thermal stability, accompanied by pickup of significant amounts of copper from the brass and bronze and of zinc and lead from the rubbers.

# LIST OF TABLES

# ILLUSTRATION

vi

# TABLE OF CONTENTS

v

# SECTION I

## INTRODUCTION

High-quality hydrocarbon fuels for aerospace applications are often affected adversely by deterioration during normal storage. Such deterioration most frequently shows up as a drop in thermal stability*. This has been a serious problem in the case of JP-6 fuel (MIL-J-25656B), "JPTS" thermally stable fuel (MIL-F-25524A), and other similar fuels. A great deal of work has been done on this problem, and various measures have been developed for preventing or correcting the deterioration. These measures include the use of special refining techniques, the use of additives, proper selection of storage system and storage conditions, and reclamation of degraded fuels by adsorptive treatment.

Fuels such as JP-6 and JPTS have presented the greatest problem in storage/thermal stability. These are fuels of intermediate thermal stability, i.e., better than the current large-volume fuels JP-4 and JP-5, but inferior to certain special fuels that can be produced at relatively high cost. The latter class of high-quality fuels includes near-pure hydrocarbons as well as naphthenic or paraffinic hydrocarbon mixtures obtained by special processing. As a convenient definition, one may describe the classes of fuels in terms of thermal stability as follows·

| | |
|---|---|
| Current large-volume fuels | 300°F and higher |
| Improved fuels (JP-6, JPTS) | 400°F and higher |
| Fuels for advanced applications | 500°F and higher |

It is generally true that the "advanced" fuels are less subject to deterioration in normal storage than are the "improved" fuels. However, this situation may be influenced by the greater precautions taken in handling the advanced fuels. Fuel contamination is minimized by rigorous precautions, and the storage and handling systems may be designed to exclude materials known to have adverse effects on fuel stability. At present, there is not a great deal of field experience in bulk handling of advanced fuels, and the available information may be further limited by considerations of military security.

In designing or adapting facilities and equipment for handling advanced fuels, it would be desirable to have firm criteria defining the materials of construction that can be tolerated and those that must be excluded. It is

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* The term "thermal stability" of jet fuels, as used in this report, refers to the resistance of the fuel to oxidative degradation at high temperatures. Although this terminology has been established by long usage, the property would be termed more correctly "thermal-oxidative stability."

1

widely known that dissolved copper in jet fuels has an extremely adverse effect on thermal stability, and therefore it is desirable to exclude or at least minimize the use of copper-base alloys in fuel handling systems. It is also considered desirable to eliminate so far as possible the use of bare steel equipment, primarily from the standpoint of minimizing contamination by rust. Many other metals and nonmetals find use in fuel handling facilities and equipment, and there is little or no information available on the effects of most of these materials on fuel thermal stability.

The results reported herein represent a preliminary examination of the effects of typical ground-fuel-system materials on the thermal stability of a naphthenic hydrocarbon fuel. The study consisted of a series of static storage tests in 55-gallon lined drums containing various material specimens. The storage temperature was 130°F, and the storage periods at the times of sampling ranged from 16 weeks to one year. This type of study serves primarily to point out materials that can be damaging to fuel thermal stability. It does not give specific information on the need for absolute exclusion of a given material from the fuel handling system. Such information can be obtained only from more elaborate static and/or dynamic exposure testing under a variety of conditions and by tests in actual or simulated fuel handling systems.

# SECTION II

# TEST MATERIALS

## A.  Test Fuel

The test fuel was a "naphthenic" type of low-volatility fuel (75 luminometer number) that is under consideration for use in high-performance aircraft. The specification covering this type of fuel at the time of procurement was MIL-T-38219 (USAF), December 1965 (Proposed), Turbine Fuel, Low Volatility. This fuel has been termed unofficially "JP-7," and this designation is used throughout this report.

The test fuel was furnished as a single 21-drum batch from one supplier. This batch of fuel, after mixing, was loaded by the supplier directly into the 55-gallon drums used in the subsequent storage test. Each drum was prerinsed with test fuel, filled, and nitrogen blanketed. The drums were filled by the supplier in April 1966, and the actual storage test was started in May 1966. All drums of fuel were held in cold-room storage until actually placed in test.

The prior history of this batch of fuel is known only in general terms. It is understood that the fuel had been produced several months previously and that reprocessing, presumably clay finishing, was necessary to restore it to specification quality. This reprocessing was performed very shortly before the product was loaded into the drums; therefore, the product used in the subsequent storage test may be considered as equivalent to freshly produced material.

The only additive present in the fuel was the antioxidant 2,6-di-tert-butyl-4-methylphenol, which was added by the supplier in amount of 4 lb/1000 bbl. The test fuel did not contain any anti-icing additive. Although the proposed MIL-T-38219 specification calls for anti-icing additive, this particular batch of fuel was purchased and tested without anti-icing additive, upon the advice of the Air Force project engineer.

Specification test data on this fuel listed in Table 1 and additional test data in Table 2 indicate conformance to the specification and reasonably good agreement among the check tests on the individual drums of fuel. Some of the divergences in flash point and distillation are slightly in excess of the ASTM precision limits. API gravities on each drum of fuel, not shown in table, were all within range of 46.0 to 46.4. The thermal stability specification limits and supplier's data are based on modified fuel coker tests with bulk-fuel heating; hence, these values are not directly comparable to the helium drive coker tests used throughout this program. As a matter of interest, however, it may be noted that the supplier's data

# TABLE 1. INSPECTION DATA ON FRESH JP-7 FUEL

| | Specs[a] | Supplier's data on composite sample | SwRI data Composite sample | SwRI data Drum No. 1 |
|---|---|---|---|---|
| Distillation: IBP, °F | 375 Min | 388 | 388 | 388 |
| 10%, °F | 400 Min | 400 | 404 | 403 |
| 20%, °F | 402 Min.[b] | 415[b] | 413 | 409 |
| 50%, °F | 420 Min. | 421 | 428 | 426 |
| 90%, °F | 500 Max. | 454 | 467 | 466 |
| EP, °F | 550 Max. | 484 | 496 | 493 |
| Residue, % | 1.5 Max. | 1.0 | 1.4 | 1.3 |
| Loss, % | 1.5 Max. | 0.0 | 0.1 | 0.1 |
| Gravity, API/60°F | 44-50 | 46.4 | --- | 46.3 |
| Existent gum, mg/100 ml | 5.0 Max. | 0.4 | | 0.4 |
| Total potential residue, 16-hr, mg/100 ml | 10.0 Max. | --- | --- | 0.4 |
| Sulfur, % | 0.1 Max. | 0.012 | --- | 0.009 |
| Mercaptan sulfur, % | 0.001 Max. | <0.0006 | --- | 0.000 |
| Aromatics, vol.%(FIA) | 5 Max. | 0.0 | --- | 1.8 |
| Copper corrosion at 212°F | 1-b Max. | 1-a | --- | 1-a |
| Flash point (P-M), °F | 150 Min. | 166 | 170 | 166 |
| Freezing point (D 2386), °F | -56 Max. | -56 | --- | -55[c] |
| Viscosity at -30°F, cs | 15 Max. | 11.9 | --- | --- |
| H$_c$, net, btu/lb | 18,750 Min. | 18,793 | --- | --- |
| btu/gal | 124,000 Min. | 124,447 | --- | --- |
| Luminometer no. | 75 Min. | 79.7 | --- | --- |
| Water reaction (interface) | 1-b Max. | 1 | --- | --- |
| Water separometer, WSIM | 85 Min. | 89 | --- | 98 |
| Thermal stability: | | | | |
| Pressure drop, in. Hg | 3 Max.[d] | 0[e] | --- | --- |
| Preheater color rating | <3[d] | 1[e] | --- | --- |
| Anti-icing additive, vol. % | 0.10-0.15 | None | None[f] | --- |
| Other additives | g | h | --- | --- |
| Particulate matter, mg/gal: | | | | |
| F.o.b. origin | 1.0 Max | 0.5 | --- | --- |
| F.o.b. destination | 4.0 Max | --- | --- | --- |

a. MIL-T-38219 (USAF), Dec 65 (Proposed), Turbine Fuel, Low Volatility.
b. Corrected for emergent stem.
c. Method D-1477.
d. Modified fuel coker, prestress 3 hr at 300°F, test at 600/600/2.5.
e. Modified fuel coker, reservoir at 300°F, test at 300/575/675/2.5.
f. Actual determination indicated 0.002%.
g. May contain 8.4 lb/Mbbl antioxidant and 2 lb/Mbbl metal deactivator.
h. Contains 4 lb/Mbbl antioxidant (2,6-di-tert-butyl-4-methylphenol).

4

# TABLE 2. ADDITIONAL INSPECTION DATA ON FRESH JP-7 FUEL

|  | Supplier's data on composite sample | SwRI data on Drum No. 1 |
|---|---|---|
| **Tests not included in specification:** | | |
| Olefins, vol. % (FIA) | 2.7 | 0.6 |
| Smoke point, mm | 39 | 32 |
| Aniline point, °F | 172.5 | |
| Aniline-gravity product | 8004 | |
| Vapor pressure, psia at 300°F | 2.4 | |
| psia at 500°F | 40.5 | |
| Specific heat at 300°F | 0.605 | |

Comparison of distillation and flash data:

| | Flash, P-M, °F | IBP, °F | 10%, °F | 20%, °F | 50%, °F | 90%, °F | EP, °F | Res., % | Loss, % |
|---|---|---|---|---|---|---|---|---|---|
| **Supplier's data on composite sample** | 166 | 383 | 400 | 405 | 421 | 454 | 484 | 1.0 | 0.0 |
| **SwRI data:** | | | | | | | | | |
| Composite sample | 170 | 388 | 404 | 413 | 428 | 467 | 496 | 1.4 | 0.1 |
| Drum No. 1 | 166 | 398 | 403 | 409 | 426 | 466 | 493 | 1.3 | 0.1 |
| Drum No. 2 | 165 | 386 | 404 | 410 | 428 | 466 | 492 | 1.3 | 0.7 |
| Drum No. 3 | 157 | 388 | 406 | 412 | 426 | 464 | 490 | 1.3 | 0.7 |
| Drum No. 6 | 161 | 388 | 408 | 414 | 426 | 460 | 488 | 1.3 | 0.7 |
| Drum No. 7 | 165 | 388 | 402 | 406 | 420 | 464 | 487 | 1.6 | 0.4 |
| Drum No. 8 | 164 | 386 | 404 | 410 | 427 | 462 | 490 | 1.5 | 0.0 |
| Drum No. 9 | 162 | 388 | 403 | 409 | 426 | 464 | 492 | 1.0 | 0.0 |
| Drum No. 10 | 162 | 388 | 402 | 409 | 426 | 463 | 498 | 1.5 | 0.5 |
| Drum No. 11 | 162 | --- | --- | --- | --- | --- | --- | --- | --- |
| Drum No. 12 | 162 | 387 | 403 | 409 | 427 | 466 | 501 | 1.2 | 0.3 |

5

indicated satisfactory thermal stability in a test with the reservoir heated to 300°F and with a 575°F preheater temperature. Thermal stability breakpoints for the fresh fuel, based on SwRI helium-drive coker results, ranged from 625° to over 675°F preheater temperature.

**B.    Test Drums**

Epoxy-lined drums were obtained on special procurement for this program. These were 55-gallon 16-gage tight-head steel drums (2-in. and 3/4-in. bungs) lined with two coats of No. 973 baked epoxy finish. This is the same type of drum used for storage of reference fuels in the CRC fuel bank. Test drums were shipped direct from the manufacturer to the refinery supplying the fuel, where they were filled with test fuel and reshipped to Wright-Patterson AFB for the test program.

**C.    Test Specimens**

Test specimens used in the storage program included typical materials encountered in present-day fuel handling systems, selected after consultation with Air Force personnel (SEG-SEMSF).

Bare and coated metal specimens were 36 in. long (round or flat) with a surface area of 115 in². Nonmetal specimens included three synthetic rubbers, a sectioned filter-separator element, and a sample of plug valve grease.

For "wet" storage conditions, distilled water in amount of 0.05% by volume was added to the contents of certain drums.

Details on the material specimens and methods of preparation are given in the following paragraphs:

Steel: Low-carbon cold-rolled mild steel, 1.5 × 36 × 0.093 in., deburred, degreased, sandblasted, and recleaned with mineral spirits.

Rusted steel: Specimens same type and size, rusted by spraying with salt solution intermittently for several weeks, wirebrushed to remove loose rust, and rinsed with water to remove salt.

Stainless steel: Type 304, cold-rolled, 1.5 × 36 × 0.095 in., deburred and then cleaned with mineral spirits*.

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* Resistant stains were removed with fine aluminum oxide abrasive paper when necessary, but no attempt was made to resurface or grind out pits and scratches.

Aluminum: Alloys 5052-H32 and 6061-T6, cold-rolled, 1.5 X 36X 0.095 in., deburred and then cleaned with mineral spirits*.

Brass: Alloy CA 360 (free-cutting yellow brass), cold-rolled, 1.5 X 36 X 0.095 in., deburred and then cleaned with mineral spirits*.

Bronze: SAE-660 bearing bronze, centrifugally cast, 1 in. diameter X 36 in. long, machined to remove outer discolored layer and then cleaned with mineral spirits.

Coated steel: Clean, sandblasted steel specimens, prepared as described previously, were coated with MIL-C-4556B (USAF) materials in accordance with the directions of the respective manufacturers. After application of the final coat, each specimen was air-cured for a minimum of seven days before installing in the drum of test fuel. The two coatings selected were representative of the two principal types of materials qualified at that time for Air Force use as fuel-tank interior coatings. These two types are identified in this report as "M" (furan) and "N" (epoxy).

Rubber specimens: These included pipe-coupling gaskets and two liners from fuel hose. All were butadiene-acrylonitrile rubbers of the Buna N type. The hose liners were low-temperature plasticized rubbers from MIL-H-26521C (USAF) hose from two different manufacturers. Liner "A" was a 36-in. length of liner from 3-in. hose, obtained by stripping the liner from completely cured hose. Liner "B" was a 36-in. length of liner for 2-in. hose, obtained by curing separately. Each of the liners was cut lengthwise into two pieces before introducing both pieces into the fuel drum. The coupling-gasket specimen consisted of two commercial fuel-resistant gaskets for 8-in. pipe. All rubber specimens were prepared by washing in water and alcohol to remove dust and other surface contaminants. The surface areas of the rubber specimens were as follows:

| | |
|---|---|
| Hose liner "A" | 593 in$^2$ |
| Hose liner "B" | 398 in$^2$ |
| Coupling gaskets | 260 in$^2$ |

Filter-separator element: An element conforming to MIL-F-52308 was sectioned to permit insertion of the pieces into the drum through the 2-in. bung. Materials of the element included polyester resin, Buna N rubber, fiberglass with phenolic resin binder, aluminum, cotton, and vinyl-coated screen. The bonded fiberglass was the major constituent in terms of surface area. The Buna N rubber was present in the form of two O-rings, having a total surface of 2.4 in$^2$.

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* Resistant stains were removed with fine aluminum oxide abrasive paper when necessary, but no attempt was made to resurface or grind out pits and scratches.

Plug valve grease: A sample of MIL-G-6032B Type I plug valve grease was obtained from one of the qualified suppliers. This product consists of a lithium stearate soap-thickened grease containing high molecular weight synthetic complex esters, with a small percentage of an aromatic amine additive. For use as a test specimen, approximately 4 oz of the grease was cut into small pieces and put into the drum of test fuel.

# SECTION III

## TEST EQUIPMENT AND PROCEDURES

### A. Storage Facilities

A cold storage room (40°F) at Wright-Patterson AFB was used to hold test fuel prior to starting the hot-storage test and to retain samples after completion of hot storage.

A hot-storage room at Wright-Patterson AFB, normally held at 130°F, was used to store drums of fuel in this program. Because of a number of malfunctions of the heating and control equipment, the hot room was unheated for rather long periods and also underwent a brief period of overheating, with temperatures near 185°F being recorded for about 2.5 hours. A summary of the hot-room temperature record is given in Table 3.

### B. Fuel Coker Equipment and Procedures

All thermal stability determinations in this program were conducted with a semiautomatic fuel coker using the "modified" test section provided by the manufacturer in accordance with CRC requirements. The fuel coker was further modified to use helium pressure rather than a pump to feed the fuel. The flow plan of the helium-drive coker is shown in Figure 1. The apparatus and procedure have been described in detail previously*.

The only operating problem encountered consistently with this equipment was "necking down" of the hot end of the heater tube after repeated testing. Tubes were originally 0.623-0.625 in. in diameter with a few tubes measuring slightly above or below these limits. They were measured after every test and discarded whenever the hot-end diameter had decreased below 0.615 inch. Application of this criterion permitted the use of a given tube for some 15 to 35 tests, depending on the severity of test conditions. This represented a reasonable compromise between the desired close dimensional control and practical considerations. With the application of this criterion, assuming a constant outer tube I.D. of 0.647 in., the radial clearance for the annulus at the hot end of the tube varied typically from 0.011 in. for a new tube to 0.016 in. for a tube at the time of discard.

The test procedure followed the general plan of ASTM D 1660-64. The test fuel samples were subjected to normal aeration and filtration through

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* Johnston, R. K. and Anderson, E. L. (Southwest Research Institute), "Effect of Additives on the Storage Stability of High-Temperature Fuels," Air Force Aero Propulsion Lab. Rept. No. APL-TR-64-142, AD-454, 998, December 1964.

9

## TABLE 3. HOT-ROOM TEMPERATURE RECORD

| Period beginning | Temp, °F [a] | Period beginning | Temp, °F [a] | Period beginning | Temp, °F [a] |
|---|---|---|---|---|---|
| 9 May 66 | 124 | 4 Nov 66 | 124 | 8 May 67 | 120 |
| 23 May 66 | 78-125[b] | 7 Nov 66 | 125 | 12 May 67 | 65[b] |
| 31 May 66 | 133-136 | 28 Nov 66 | 125 | 15 May 67 | 65[b] |
|  |  |  |  | 30 May 67 | 85[b] |
| 6 Jun 66 | 133-142 | 5 Dec 66 | 125 |  |  |
| 14 Jun 66 | 130-138 | 12 Dec 66 | 125 | 2 Jun 67 | 90 |
| 20 Jun 66 | 135-145 | 17 Dec 66 | 125 | 6 Jun 67 | 130 |
| 27 Jun 66 | 130-145 |  |  | 9 Jun 67 | 140-142 |
|  |  | 3 Jan 67 | 125 | 14 Jun 67 | 100[b] |
| 5 Jul 66 | 90-110 | 9 Jan 67 | 125 | 19 Jun 67 | 100[b] |
| 11 Jul 66 | 85-90 [b] | 16 Jan 67 | 125 | 26 Jun 67 | 100[b] |
| 18 Jul 66 | 82-130[b] | 23 Jan 67 | 125 |  |  |
| 25 Jul 66 | 62-126[b] | 30 Jan 67 | 125 | 3 Jul 67 | 100[b] |
|  |  |  |  | 10 Jul 67 | 98[b] |
| 8 Aug 66 | 88-120 | 6 Feb 67 | 125 | 17 Jul 67 | 96[b] |
| 23 Aug 66 | 122-135 | 13 Feb 67 | 105 | 26 Jul 67 | 101[b] |
| 30 Aug 66 | 185[c] | 20 Feb 67 | 60[b] | 31 Jul 67 | 99[b] |
|  |  | 23 Feb 67 | 60[b] |  |  |
| 16 Sep 66 | 120-145 |  |  | 7 Aug 67 | 100[b] |
| 26 Sep 66 | 122-128 | 6 Mar 67 | 123 | 14 Aug 67 | 92[b] |
|  |  | 20 Mar 67 | 130 | 21 Aug 67 | 85[b] |
| 3 Oct 66 | 123-128 |  |  |  |  |
| 17 Oct 66 | 120 | 17 Apr 67 | 130 | 11 Sep 67 | 92[b] |
| 21 Oct 66 | 125 | 24 Apr 67 | 130 |  |  |

a. Temperatures shown as ranges are taken from recorder chart for period indicated. Individual temperatures are spot readings.

b. Heating system out of service for most or all of indicated period.

c. Temperature of 185°F was recorded for about 2.5 hours on 30 Aug 66, after which heating unit was shut down for 2-3 days and then restored to normal operation.

10
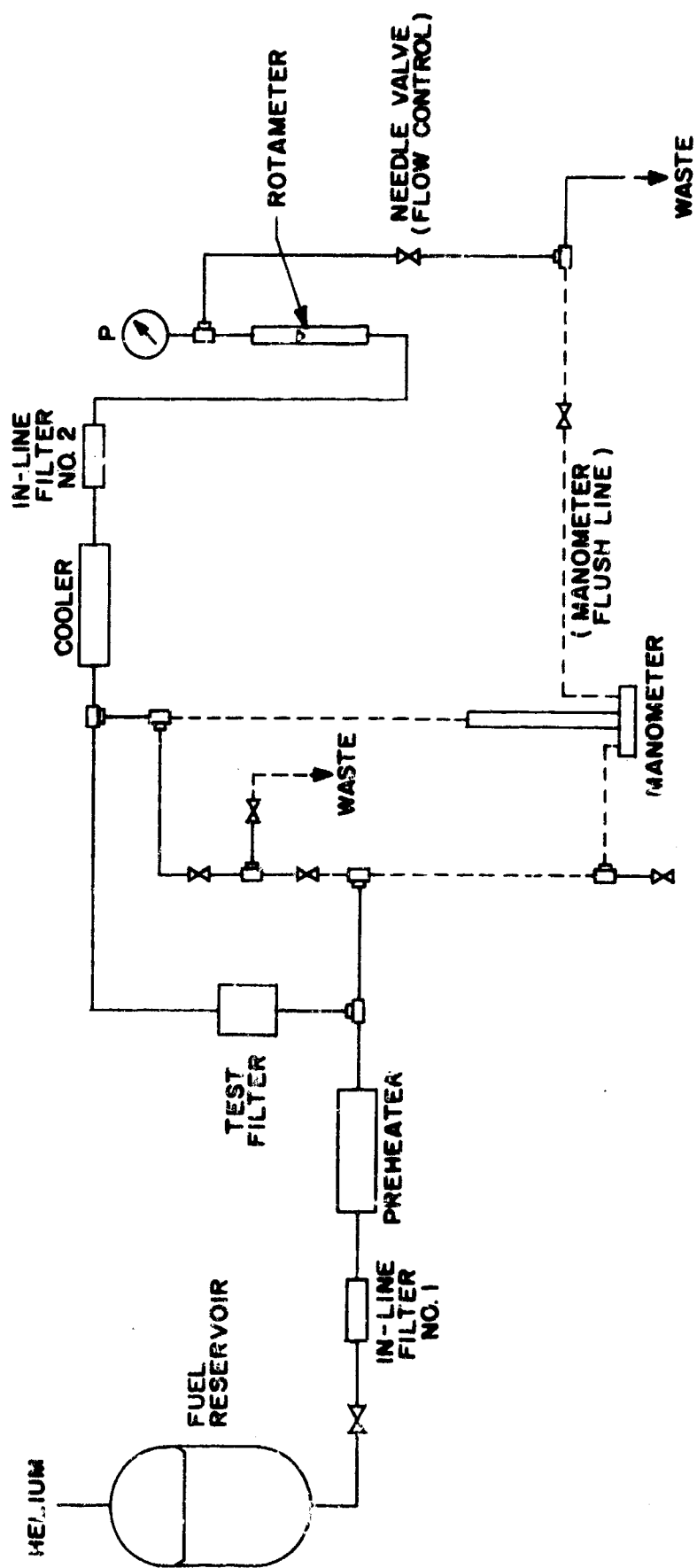
FIGURE 1. SwRI MODIFIED FUEL COKER WITH HELIUM DRIVE

11

No. 12 Whatman paper prior to test. No bulk-fuel preheating was employed. Helium drive pressure was 250 psig, and fuel flow rate was 2.5 lb/hr. The filter temperature was maintained 100°F above the preheater fuel-out temperature, except that the filter temperature was limited to a maximum of 700°F to prevent the possibility of boiling within the filter. The highest preheater fuel-out temperature that could be attained and controlled properly was 675°F. Thus, the filter temperature was 700°F for preheater temperatures of 600 to 675°F, inclusive.

Series of tests were run on each fuel sample at intervals of 25°F in preheater fuel-out temperature. Failure was defined as a maximum preheater color rating (unwiped) of No. 3 or darker, or a filter pressure drop of 2 in. Hg or more during the 300-minute test. The "breakpoint" was defined as the lowest preheater fuel-out temperature giving failure by either of these criteria. Thus, the breakpoint was nominally established to the nearest 25°F but is considered significant only to the nearest 50°F because of the well-known problems with precision of fuel coker results in general.

Preheater colors were rated using the "Tuborator" furnished by the manufacturer of the coker. Color ratings from 0 to 4 were based on the official ASTM scale (D 1660), and darker colors were estimated using the 0 to 8 scale developed in early CRC work on the research fuel coker. It should be noted that many of the preheater tubes were very difficult to rate properly, since the colors encountered frequently did not match the shades of the color standards. This is a well-known problem and is nothing unique to this particular program. Color ratings obtained in this program are considered to be reasonably self-consistent but not necessarily indicative of how the same tubes would be rated by other laboratories.

All preheater tubes were rated both in the unwiped and wiped conditions; the unwiped ratings were used in determining breakpoints. Although the use of the helium-drive coker eliminates the glaring discrepancies often caused by pump wear debris, there were instances in this program when wipable deposits were encountered in sufficient amounts to give appreciable differences in ratings. In such cases, the wiping usually lowered the preheater color rating, but there were a few cases in which the wiping removed a light-colored deposit, revealing a darker deposit on the tube.

No formal study was made of the repeatability of results in this program. Duplicate tests were run at or near the breakpoint temperature whenever possible. The repeatability was satisfactory in most cases. A discussion of these check-test results is given in Section IV. In this program, results from duplicate tests were not averaged. Instead, the "worst" (most severe) result was used in establishing the breakpoint. The choice of criteria, i.e., worst result vs. average, would not affect the breakpoint by more than 25°F in any case.

12

One set of check tests with another laboratory was performed in order to obtain a comparison of severity level. The test fuel was a thermally stable fuel from another storage program. Thermal stability tests were run by the University of Dayton Research Institute (UDRI), using helium-drive coker equipment similar (but not identical) to that used by SwRI. The following results were obtained:

| Preheater/filter temperature, °F: | 650/675 | 675/675 |
|---|---|---|
| UDRI: Preheater color, unwiped (wiped) | 2(1) | 3(2) |
| Filter $\Delta P$, in. Hg | 0 | 0.4 |
| SwRI: Preheater color, unwiped (wiped) | 2(2) | 6(6) |
| Filter $\Delta P$, in. Hg | 0 | 0 |

It will be noted that both laboratories obtained a breakpoint of 675°F on this fuel. This single comparison indicates good agreement between the two laboratories but of course does not give any quantitative measure of reproducibility. The SwRI tests were somewhat more severe than the UDRI tests in preheater color rating, but this could well reflect individual-test variations rather than any significant difference in severity level between the two laboratories.

C.    Analyses for Metal Contents

Analyses for copper, iron, lead, and zinc contents of the test fuels were performed by Monsanto Research Corporation under Air Force Contract No. AF 33(615)-3277. The methods for copper, iron, and zinc have been given in a report by Lander*, and the method for iron has been discussed in more detail in a report by Scribner, et al.** The method for lead was developed by Monsanto Research Corporation in connection with the determinations reported herein and has been described briefly in a private communication. All methods are designed for determination of metal contents down to a few parts per billion and are based on extraction of the metal, complexing, and spectrophotometric determination. The chromogenic agents are as follows:

| | |
|---|---|
| Copper | Zinc dibenzyldithiocarbamate |
| Iron | Bathophenanthroline |
| Lead | Dithizone |
| Zinc | Dithizone |

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*    Lander, H. R., Jr., "Stability of High-Temperature Hydrocarbon Jet Fuels During Storage, " Air Force Aero Propulsion Lab. Rept. No. APL-TDR-64-107, November 1964.

**   Scribner, W. G., et al. (Monsanto Research Corp.), "Determination of Parts per Billion Iron in Hydrocarbon Jet Fuels, " Air Force Aero Propulsion Lab. Rept. No. AFAPL-TR-66-44, April 1966.

The lower limits of detection (as given in a private communication from Dr. W. G. Scribner of Monsanto Research) re 5 parts per billion for copper, iron and zinc, and 10 parts per billion for lead; the accuracy of the determinations is estimated to be within 20%. For the copper, iron, and zinc methods, completeness of extraction and noninterference of allowed fuel additives and other metals normally present have been established experimentally. For the lead determination, such verifications have not been made, and it is considered probable that the method is not applicable when trace amounts of lead are present as lead alkyl compounds.

D.    Sample Storage and Handling Procedures

All drums of test fuel were held in cold storage until ready for the start of hot-room storage. Two drums (Nos. 1 and 21 in the filling sequence of supplier) were kept in cold storage for thermal stability and inspection tests, and the other 19 drums were transferred to hot room storage. The fuel storage conditions were as follows:

| | |
|---|---|
| Storage temperature | 130°F |
| Storage container | 55-gal drum, epoxy lined |
| Initial fuel volume | 45 gal |
| Fuel sampling | 15 gal each at 16, 35, and 52 weeks |
| Air availability | Each drum aerated prior to storage and at each intermediate sampling; drums kept sealed during storage |
| Moisture availability | Fuel stored either "dry" (as received) or "wet" (0.05% distilled water added prior to hot storage) |

The initial thermal stability breakpoint was determined on each drum of fuel at the time it was placed in hot-room storage, and the breakpoints were also determined after 16, 35, and 52 weeks of storage.

The drums of fuel were placed in hot-room storage on a schedule of one drum each week. This staggered schedule permitted the fuel coker testing to keep pace with the storage and sampling schedule; i.e., there was no pileup of samples or delay in running the thermal stability tests on either the initial or stored samples. This scheduling was considered desirable to eliminate any possibility of changes in fuel properties during an interim holding period. However, the staggered schedule, plus the frequent malfunctions of the hot room, did introduce drum-to-drum differences in storage history.

14

The starting sequence for each drum placed in hot storage was as follows:

Remove from cold storage and bring to ambient temperature.
Draw off enough fuel to leave 45 gallons in drum (the fuel removed
    is used for initial thermal stability determinations).
Aerate fuel in drum by blowing with dry air.
Add water (if applicable).
Add material specimen.
Bung tight and transfer to hot storage.

The intermediate sampling sequence for each drum was as follows:

Remove from hot storage and bring to ambient temperature.
Draw off 15 gallons of fuel.
Aerate fuel remaining in drum by blowing with dry air.
Bung tight and return to hot storage.

At the final 52-week sampling, the drums were cut open for inspection
of material specimens and drum interiors.

# SECTION IV

## TEST RESULTS AND DISCUSSION

**A.** Thermal Stability Test Results

Complete fuel coker test results are listed in Table 4. These test series will not discussed individually. However, a few important points that are not evident from the subsequent summary table will be discussed here.

The fresh fuels before hot-room storage did not show any evidence of filter plugging in the fuel coker tests, and their preheater breakpoints ranged from 625°F to above 675°F. The fuels after hot-room storage showed a fairly high incidence of filter plugging in the fuel coker tests; the breakpoints ranged from below 375°F to above 675°F. Filter plugging usually became more pronounced as hot-room storage time increased, in those few cases in which directly comparable data are available. The filter plugging was not confined to fuels with any given type or types of material specimens. Noteworthy, however, is the very severe filter plugging encountered at low test temperatures with the three fuels that had been stored with synthetic rubber specimens (Drums 11, 13, and 18).

Whenever possible, duplicate test results were obtained on each fuel sample at or near its breakpoint. This was often infeasible because of sample limitations and difficulty in pinpointing the failure range of a given sample with a minimum number of tests. However, there were 24 fuel samples for which such data are available on preheater breakpoints; these data, which are shown in Table 5, give an indication of test repeatability. For 19 out of the 24 samples, the replicate tests defined the breakpoint satisfactorily without any repeatability problem. For the remaining 5 samples, the replicate test results "straddled" the breakpoint criterion; i.e., one rating was 3 or higher and the other was 2 or lower. The deviations were mostly only 1 or 2 color-code units, with one instance of a large deviation (Drum 1, fresh). Inspection of the preheater ratings listed in Table 5 indicates that the breakpoint would not be shifted by more than 25°F by choice of high or low rating.

The repeatability of filter plugging ratings at the breakpoint is illustrated by the data shown in Table 6, which include only those cases in which significant filter plugging was observed at the breakpoint. It will i noted that the repeatability of filter plugging under these conditions was generally very poor; in most of the cases, the two test results "straddled" the breakpoint criterion of 2.0 in. Hg. However, it will also be noted that this poor repeatability of filter plugging did not affect the overall breakpoint in most cases, since preheater failures also governed the breakpoint. In only two cases, the use of a single, nonrepeatable filter rating shifted the

16

## TABLE 4. HELIUM-DRIVE COKER RESULTS ON FUELS FROM HOT STORAGE

All temperatures shown are preheater fuel-out temperatures, °F.
Filter pressure drops, in. Hg, refer to end of 300-min test unless shorter
  period (min) is shown in parentheses.
Preheater color ratings are unwiped (wiped ratings in parentheses).
Breakpoint is lowest temperature giving an unwiped preheater rating of 3 or
  higher (P) or a filter pressure drop of 2 in. Hg or more (F).

| | | | | | | | | Breakpoint |
|---|---|---|---|---|---|---|---|---|
| **Drum No. 1 — No specimen, cold storage** | | | | | | | | |
| Fresh: | Temp. | 600 | 625 | 625 | 625 | 650 | 650 | 625(P) |
| | ΔP | 0 | 0 | 0 | 0 | 0 | 0 | |
| | Color | 2(1) | 5(4) | 8(8) | 2(2) | 4(2) | 5(5) | |
| After cold storage as indicated: | | | | | | | | |
| | Weeks | 15 | 34 | 54 | | | | |
| | Temp. | 625 | 600 | 625 | | | | |
| | ΔP | 0 | 0 | 0 | | | | |
| | Color | 2(2) | 2(2) | 4(1) | | | | |
| **Drum No. 2 — No specimen** | | | | | | | | |
| Fresh: | | Not run | | | | | | |
| 16-week: | Temp. | 575 | 600 | 625 | 625 | 625 | | 625(P) |
| | ΔP | 0 | 0 | 0 | 0 | 0 | | |
| | Color | 2 | 2(2) | 4(3) | 2(2) | 3(2) | | |
| 35-week: | Temp. | 625 | 650 | 675 | | | | 650(PF) |
| | ΔP | 0 | 2.0 | 11.5 | | | | |
| | Color | 2(2) | 4(4) | 4(5) | | | | |
| 1-year: | Temp. | 625 | 650 | | | | | 650(P) |
| | ΔP | 0.2 | 0.5 | | | | | |
| | Color | 2(2) | 4(3) | | | | | |
| **Drum No. 3 — No specimen, wet** | | | | | | | | |
| Fresh: | | Not run | | | | | | |
| 16-week: | Temp. | 625 | 675 | 675 | | | | >675 |
| | ΔP | 0 | 0 | 0.2 | | | | |
| | Color | 2(2) | 2(2) | 2(2) | | | | |
| 35-week: | Temp. | 575 | 600 | 625 | 625 | | | 625(P) |
| | ΔP | 0 | 0 | 0 | 0 | | | |
| | Color | 2(2) | 2(2) | 5(5) | 4(4) | | | |
| 1-year: | Temp. | 525 | 550 | 600 | 625 | | | 550(P) |
| | ΔP | 0 | 0 | 0 | 0.2 | | | |
| | Color | 1(1) | 3(2) | 4(3) | 3(2) | | | |

17

# TABLE 4. HELIUM-DRIVE COKER RESULTS ON FUELS FROM HOT STORAGE (Contd.)

Breakpoint

### Drum No. 4 — Type 304 stainless steel

| Fresh: | Temp. | 625 | 675 | 675 | | | | >675 |
|---|---|---|---|---|---|---|---|---|
| | ΔP | 0 | 0 | 0 | | | | |
| | Color | 2(2) | 2(2) | 2(2) | | | | |
| 16-week: | Temp. | 625 | 650 | 675 | 675 | 675 | | 675(P) |
| | ΔP | 0.2 | 0 | 0 | 0 | 0 | | |
| | Color | 2(2) | 2(2) | 4(4) | 4(3) | 3(3) | | |
| 35-week: | Temp. | 625 | 650 | 650 | 675 | | | 650(P) |
| | ΔP | 0 | 0.4 | 1.5 | 10.8 | | | |
| | Color | 2(1) | 4(4) | 3(3) | 4(4) | | | |
| 1-year: | Temp. | 525 | 575 | 575 | 600 | 600 | 625 | 575(P) |
| | ΔP | 0 | 0 | 0 | 0 | 3.0 | 0 | |
| | Color | 1(1) | 1(1) | 3(3) | 3(3) | 3(4) | 5(5) | |

### Drum No. 5 — 6061 aluminum

| Fresh: | Temp. | 625 | 675 | | | >675 |
|---|---|---|---|---|---|---|
| | ΔP | 0 | 0 | | | |
| | Color | 2(2) | 2(2) | | | |
| 16-week: | Temp | 625 | 650 | 675 | 675 | 675(P) |
| | ΔP | 0.1 | 0 | 0 | 0 | |
| | Color | 1(1) | 2(2) | 3(3) | 3(3) | |
| 35-week: | Temp. | 575 | 600 | 625 | 625 | 625(PF) |
| | ΔP | 0 | 0 | 0.3 | 21.7 | |
| | Color | 1(1) | 2(2) | 3(3) | 2(2) | |
| 1-year: | Temp. | 575 | 600 | 600 | 625 | 600(P) |
| | ΔP | 0 | 0 | 0 | 29.8 | |
| | Color | 2(1) | 3(3) | 3(3) | 5(5) | |

### Drum No. 6 — 5052 aluminum

| Fresh: | Temp. | 625 | 675 | 675 | | >675 |
|---|---|---|---|---|---|---|
| | ΔP | 0 | 0 | 0 | | |
| | Color | 1(1) | 2(2) | 0 | | |
| 16-week: | Temp. | 625 | 650 | 675 | 675 | 675(P) |
| | ΔP | 0 | 0 | 0 | 0 | |
| | Color | 1(1) | 2(2) | 3(3) | 2(3) | |
| 35-week: | Temp. | 625 | 650 | 675 | 675 | 675(PF) |
| | ΔP | 0 | 0 | 0 | 6.9 | |
| | Color | 1(1) | 2(2) | 5(5) | 5(5) | |
| 1-year: | Temp. | 575 | 600 | 600 | 625 | 600(PF) |
| | ΔP | 0 | 2.4 | 0.1 | 0 | |
| | Color | 1(1) | 3(2) | 5(3) | 4(3) | |

18

## TABLE 4. HELIUM-DRIVE COKER RESULTS ON FUELS FROM HOT STORAGE (Contd.)

**Drum No. 7 — CA 360 brass**

| Fresh: | | | | | | | >675 |
|---|---|---|---|---|---|---|---|
| | Temp. | 625 | 675 | | | | |
| | ΔP | 0 | 0.2 | | | | |
| | Color | 0 | 2(2) | | | | |
| 16-week: | Temp. | 525 | 550 | 575 | 575 | 625 | 575(P) |
| | ΔP | 0.2 | 0 | 0 | 0.2 | 0.2 | |
| | Color | 1(1) | `.) | 3(3) | 3(3) | 4(3) | |
| 35-week: | Temp. | 375 | 425 | 475 | 525 | 575 | ≤375(PF) |
| | ΔP | 26(212) | 0.3 | 0.1 | 0.1 | 0 | |
| | Color | 6(4) | 3(3) | 3(3) | 3(3)[a] | 3(3) | |
| 1-year: | Temp. | 400 | 450 | 500 | 525 | 550 | 525(P) |
| | ΔP | 0.2 | 0.1 | 0 | 0 | 0 | |
| | Color | 1(1) | 1(1) | 2(2) | 3(3) | 5(5) | |

**Drum No. 8 — CA 360 brass, wet**

| Fresh: | Temp. | 600 | 625 | 625 | | | 625(P) |
|---|---|---|---|---|---|---|---|
| | ΔP | 0 | 0 | 0 | | | |
| | Color | 2(2) | 4(4) | 4(4) | | | |
| 16-week: | Temp. | 475 | 475 | 525 | 575 | 625 | ≤475(P) |
| | ΔP | 0.2 | 0 | 0 | 0.2 | 0 | |
| | Color | 4(1) | 4(3) | 4(3) | 4(3) | 4(4) | |
| 35-week: | Not run | | | | | | |
| 1-year: | Temp. | 475 | 500 | 500 | 525 | | 525(P) |
| | ΔP | 0 | 0 | 2.6[b] | 0 | | |
| | Color | 1(1) | 2(2) | 1(1) | 3(3) | | |

**Drum No. 9 — SAE 660 bronze**

| Fresh: | Temp. | 625 | 675 | | | | >675 |
|---|---|---|---|---|---|---|---|
| | ΔP | 0 | 0 | | | | |
| | Color | 2(2) | 2(2) | | | | |
| 16-week: | Temp. | 475 | 525 | 575 | 625 | | ≤475(F) |
| | ΔP | 0 | 0 | 0.1 | 0 | | |
| | Color | 6(5) | 6(6) | 6(6) | 5(3) | | |
| 35-week: | Not run | | | | | | |
| 1-year: | Temp. | 475 | 525 | 550 | 575 | | 550(P) |
| | ΔP | 0 | 0 | 0 | 0 | | |
| | Color | 1(1) | 2(2) | 4(3) | 5(5) | | |

a. White powdery deposit on tube; difficult to rate before wiping.

b. Result considered suspect because of bad condition of sample can.

# TABLE 4. HELIUM-DRIVE COKER RESULTS ON FUELS FROM HOT STORAGE (Contd.)

Breakpoint

### Drum No. 10 — Plug valve grease

| | | | | | | | | Breakpoint |
|---|---|---|---|---|---|---|---|---|
| Fresh: | Temp. | 625 | 675 | | | | | >675 |
| | ΔP | 0 | 0 | | | | | |
| | Color | 2(2) | 2(2) | | | | | |
| 16-week: | Temp. | 625 | 675 | 675 | | | | >675 |
| | ΔP | 0 | 0 | 0 | | | | |
| | Color | 2(2) | 2(2) | 2(2) | | | | |
| 35-week: | Temp. | 575 | 600 | 625 | | | | 600(P) |
| | ΔP | 0 | 0 | 0 | | | | |
| | Color | 1(2) | 5(3) | 6(5) | | | | |
| 1-year: | Temp. | 600 | 650 | 675 | 675 | | | 675(F) |
| | ΔP | 0 | 1.9 | 0.1 | 3.5 | | | |
| | Color | 1(1) | 1(1) | 1(1) | 2(2) | | | |

### Drum No. 11 — Hose liner "A"

| | | | | | | | | Breakpoint |
|---|---|---|---|---|---|---|---|---|
| Fresh: | Temp. | 600 | 625 | 625 | | | | 625(P) |
| | ΔP | 0.3 | 0 | 0 | | | | |
| | Color | 2(2) | 3(3) | 3(2) | | | | |
| 16-week: | Temp. | 375 | 425 | 475 | 525 | 575 | 625 | ≤375(P) |
| | ΔP | 0 | 0 | 0 | 0 | 0 | 0 | |
| | Color | 6(2) | 6(6) | 6(6) | 6(6) | 6(5) | 5(3) | |
| 35-week: | | Not run | | | | | | |
| 1-year: | Temp. | 350 | 400 | | | | | ≤350(F) |
| | ΔP | 25(175) | 25(40) | | | | | |
| | Color | 1(1) | 5(6) | | | | | |

### Drum No. 12 — F/S element

| | | | | | | | | Breakpoint |
|---|---|---|---|---|---|---|---|---|
| Fresh: | Temp. | 625 | 650 | 675 | | | | 650(P) |
| | ΔP | 0 | 0 | 0 | | | | |
| | Color | 2(2) | 4(4) | 3(2) | | | | |
| 16-week: | Temp. | 475 | 575 | 600 | 625 | 625 | | 625(P) |
| | ΔP | 0.1 | 0 | 0.2 | 0 | 0 | | |
| | Color | 1(1) | 1(1) | 2(2) | 4(4) | 5(6) | | |
| 35-week: | Temp. | 575 | 600 | 600 | 625 | | | 600(P) |
| | ΔP | 0 | 0 | 0 | 0 | | | |
| | Color | 2(2) | 3(4) | 3(3) | 3(5) | | | |
| 1-year: | Temp. | 600 | 625 | 650 | | | | 625(P) |
| | ΔP | 0 | 0.3 | 2.5 | | | | |
| | Color | 1(1) | 3(2) | 4(4) | | | | |

20

## TABLE 4. HELIUM-DRIVE COKER RESULTS ON FUELS FROM HOT STORAGE (Contd.)

Drum No. 13 — Coupling gasket

| | | | | | | | | Breakpoint |
|---|---|---|---|---|---|---|---|---|
| Fresh: | Temp. | 625 | 675 | 675 | | | | >675 |
| | ΔP | 0 | 0 | 0 | | | | |
| | Color | 2(2) | 2(2) | 2(2) | | | | |
| 16-week: | Temp. | 425 | 450 | 475 | 525 | 575 | 625 | 450(P) |
| | ΔP | 0 | 0 | 0 | 0 | 0 | 0 | |
| | Color | 1(1) | 6(6) | 6(6) | 6(6) | 6(6) | 6(6) | |
| 35-week: | Not run | | | | | | | |
| 1-year: | Temp. | 350 | 400 | 450 | | | | ≤350(P) |
| | ΔP | 0 | 22(20) | 30(30) | | | | |
| | Color | 3(2) | 5(5) | 6(6) | | | | |

Drum No. 14 — Steel

| | | | | | | | Breakpoint |
|---|---|---|---|---|---|---|---|
| Fresh: | Temp. | 625 | 675 | 675 | | | >675 |
| | ΔP | 0 | 0.2 | 0 | | | |
| | Color | 1(1) | 2(2) | 2(2) | | | |
| 16-week: | Temp. | 575 | 600 | 600 | 625 | 625 | 625(PF) |
| | ΔP | 0 | 0 | 0.5 | 0 | 7.6(210) | |
| | Color | 1(2) | 2(2) | 1(1) | 5(5) | 6(6) | |
| 35-week: | Temp. | 575 | 600 | 600 | 625 | | 600(P) |
| | ΔP | 0 | 0 | 0.6 | 0 | | |
| | Color | 1(1) | 4(4) | 3(3) | 4(3) | | |
| 1-year: | Temp. | 600 | 650 | 675 | | | 675(P) |
| | ΔP | 0 | 0.4 | 0.3 | | | |
| | Color | 2(2) | 2(2) | 4(2) | | | |

Drum No. 15 — Rusted steel

| | | | | | | | Breakpoint |
|---|---|---|---|---|---|---|---|
| Fresh: | Temp. | 625 | 675 | 675 | | | >675 |
| | ΔP | 0 | 0 | 0 | | | |
| | Color | 2(2) | 2(2) | 2(2) | | | |
| 16-week: | Temp. | 525 | 550 | 575 | 575 | 625 | 575(P) |
| | ΔP | 0 | 0 | 0 | 0 | 0 | |
| | Color | 1(1) | 2(1) | 4(2) | 4(1) | 5(5) | |
| 35-week: | Temp. | 475 | 500 | 525 | 525 | 575 | 500(P) |
| | ΔP | 0 | 0 | 0 | 0 | 0 | |
| | Color | 1(1) | 4(4) | 4(2) | 5(1) | 5(5) | |
| 1-year: | Temp. | 500 | 525 | 550 | | | 550(P) |
| | ΔP | 0 | 0 | 0 | | | |
| | Color | 1(1) | 1(1) | 3(2) | | | |

## TABLE 4. HELIUM-DRIVE COKER RESULTS ON FUELS FROM HOT STORAGE (Contd.)

Breakpoint

**Drum No. 16 — Rusted steel, wet**

| Fresh: | Temp. | 625 | 675 | 675 | | >675 |
|---|---|---|---|---|---|---|
| | $\Delta P$ | 0.4 | 0 | 0 | | |
| | Color | 2(2) | 2(2) | 2(2) | | |

| 16-week: | Temp. | 625[a] | 625 | 675 | 675 | >675 |
|---|---|---|---|---|---|---|
| | $\Delta P$ | 0 | 0 | 0.2 | 0 | |
| | Color | 3(3) | 1(1) | 2(2) | 2(2) | |

| 35-week: | Temp. | 575 | 600 | 625 | | 600(P) |
|---|---|---|---|---|---|---|
| | $\Delta P$ | 0 | 0 | 0 | | |
| | Color | 2(2) | 5(5) | 4(4) | | |

| 1-year: | Temp. | 550 | 575 | 600 | | 600(P) |
|---|---|---|---|---|---|---|
| | $\Delta P$ | 0 | 0 | 0 | | |
| | Color | 2(2) | 2(1) | 4(3) | | |

**Drum No. 17 — Coating "M"**

| Fresh: | Temp. | 625 | 675 | 675 | | >675 |
|---|---|---|---|---|---|---|
| | $\Delta P$ | 0 | 0.3 | 0 | | |
| | Color | 2(2) | 2(3) | 2(2) | | |

| 16-week: | Temp. | 625 | 650 | 650[b] | 675 | 650(P) |
|---|---|---|---|---|---|---|
| | $\Delta P$ | 0 | 0 | 0 | 0.3 | |
| | Color | 1(1) | 3(2) | 3(1) | 4(2) | |

| 35-week: | Temp. | 575 | 600 | 625 | 625 | 625(P) |
|---|---|---|---|---|---|---|
| | $\Delta P$ | 0 | 0 | 0 | 0 | |
| | Color | 1(1) | 2(2) | 4(4) | 5(5) | |

| 1-year: | Temp. | 625 | 650 | 675 | | 675(P) |
|---|---|---|---|---|---|---|
| | $\Delta P$ | 0 | 0 | 0.2 | | |
| | Color | 1(1) | 1(1) | 3(2) | | |

**Drum No. 18 — Hose liner "B"**

| Fresh: | Temp. | 625 | 650 | 675 | | 650(P) |
|---|---|---|---|---|---|---|
| | $\Delta P$ | 0.1 | 0 | 0 | | |
| | Color | 2(2) | 3(3) | 3(3) | | |

| 16-week: | Temp. | 400 | 475 | 525 | 625 | ≤400(PF) |
|---|---|---|---|---|---|---|
| | $\Delta P$ | 30(15) | 30(9) | 30(15) | 30(25) | |
| | Color | 5(5) | 6(6) | 6(6) | 6(6) | |

| 35-week: | | Not run | | | | |
|---|---|---|---|---|---|---|

| 1-year: | Temp. | 350 | 375 | 400 | | 400(PF) |
|---|---|---|---|---|---|---|
| | $\Delta P$ | 0 | 0 | 25(18) | | |
| | Color | 1(1) | 1(1) | 6(6) | | |

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a. Fuel sample cloudy after prefiltration; coker result not used in establishing breakpoint.

b. Run terminated at 140 minutes because of power failure.

22

## TABLE 4. HELIUM-DRIVE COKER RESULTS ON FUELS FROM HOT STORAGE (Contd.)

<div align="right"><u>Breakpoint</u></div>

### Drum No. 19 — Coating "N"

| | | | | | | | Breakpoint |
|---|---|---|---|---|---|---|---|
| Fresh: | Temp. | 625 | 675 | 675[a] | | | >675 |
| | ΔP | 0 | 0.2 | 0 | | | |
| | Color | 2(2) | 2(2) | 2(2) | | | |
| 16-week: | Temp. | 625 | 650 | 675 | 675 | | 675(PF) |
| | ΔP | 0 | 0 | 30(180) | 6.9 | | |
| | Color | 2(1) | 2(1) | 4(3) | 3(3) | | |
| 35-week: | Temp. | 575 | 600 | 625 | | | 625(P) |
| | ΔP | 0 | 0 | 0 | | | |
| | Color | 2(2) | 2(2) | 3(3) | | | |
| 1-year: | Temp. | 575 | 600[b] | 625 | | | 600(P) |
| | ΔP | 0 | 0 | 0 | | | |
| | Color | 2(2) | 6(6) | 2(3) | | | |

### Drum No. 20 — No specimen

| | | | | | | | Breakpoint |
|---|---|---|---|---|---|---|---|
| Fresh: | Temp. | 625 | 675 | 675 | | | >675 |
| | ΔP | 0 | 0.2 | 0 | | | |
| | Color | 1(1) | 2(2) | 2(2) | | | |
| 16-week: | Temp. | 625 | 650[c] | 650 | 675 | 675 | 650(F) |
| | ΔP | 0 | 0.9 | 2.5 | 30(240) | 4.3 | |
| | Color | 1(1) | 2(3) | 2(3) | 4(4) | 2(2) | |
| 35-week: | Temp. | 625 | 650 | 675 | | | 650(PF) |
| | ΔP | 0.2 | 4.0 | 1.9 | | | |
| | Color | 1(1) | 4(4) | 4(4) | | | |
| 1-year: | Temp. | 625 | 675 | | | | >675 |
| | ΔP | 0 | 0 | | | | |
| | Color | 2(2) | 2(2) | | | | |

### Drum No. 21 — No specimen, cold storage

| | | | | | | | Breakpoint |
|---|---|---|---|---|---|---|---|
| Fresh: | Temp. | 625 | 650 | 675 | | | >675 |
| | ΔP | 0 | 0 | 0 | | | |
| | Color | 2(2) | 2(2) | 2(2) | | | |

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a. Test terminated at 240 minutes because of instrument malfunction.

b. Test terminated at 294 minutes because of exhaustion of fuel supply.

c. Fuel sample prefiltered through 0.45 μ membrane filter.

23

# TABLE 5. PREHEATER RATINGS IN REPLICATE TESTS AT BREAKPOINT

| | Preheater ratings | | |
|---|---|---|---|
| | 25°F below breakpoint | At breakpoint | 25°F above breakpoint |
| Drum 1 (fresh) | 2 | 5, 8, 2 | 4, 5 |
| Drum 2 (16-wk) | 2 | 4, 2, 3 | |
| Drum 3 (35-wk) | 2 | 5, 4 | |
| Drum 4 (16-wk) | 2 | 4, 4, 3 | |
| Drum 4 (35-wk) | 2 | 4, 3 | $4^b$ |
| Drum 4 (1-yr) | $1^a$ | 1, 3 | 3, 3 |
| Drum 5 (16-wk) | 2 | 3, 3 | |
| Drum 5 (35-wk) | 2 | 3, $2^b$ | |
| Drum 5 (1-yr) | 2 | 3, 3 | $5^b$ |
| Drum 6 (16-wk) | 2 | 3, 2 | |
| Drum 6 (35-wk) | 2 | 5, $5^b$ | |
| Drum 6 (1-yr) | 1 | 3, $5^b$ | 4 |
| Drum 7 (16-wk) | 1 | 3, 3 | |
| Drum 8 (fresh) | 2 | 4, 4 | |
| Drum 11 (fresh) | 2 | 3, 3 | |
| Drum 12 (16-wk) | 2 | 4, 5 | |
| Drum 12 (35-wk) | 2 | 3, 3 | 3 |
| Drum 14 (16-wk) | 2, 1 | 5, $6^b$ | |
| Drum 14 (35-wk) | 1 | 4, 3 | 4 |
| Drum 15 (16-wk) | 2 | 4, 4 | 5 |
| Drum 17 (16-wk) | 1 | 3, 3 | 4 |
| Drum 17 (35-wk) | 2 | 4, 5 | |
| Drum 19 (16-wk) | 2 | 4, $3^b$ | |
| Drum 20 (16-wk) | 1 | 2, $2^b$ | 4, $2^b$ |

NOTE: This table includes only those samples for which a definite breakpoint temperature was obtained with two or more tests at the breakpoint.

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a. At 50°F below breakpoint.

b. Gave filter breakpoint; see Table 6.

# TABLE 6. FILTER AND PREHEATER RATINGS IN REPLICATE TESTS AT BREAKPOINT

| | | 25°F below breakpoint | | At breakpoint | | 25°F above breakpoint | |
|---|---|---|---|---|---|---|---|
| Drum 4 (35-wk): | $\Delta P$, in. Hg | 0 | | 0.4 | 1.5 | 10.8 | |
| | Color | 2 | | 4 | 3 | 4 | |
| Drum 5 (35-wk): | $\Delta P$, in. Hg | 0 | | 0.3 | 21.7 | | |
| | Color | 2 | | 3 | 2 | | |
| Drum 6 (35-wk): | $\Delta P$, in. Hg | 0 | | 0 | 6.9 | | |
| | Color | 2 | | 5 | 5 | | |
| Drum 6 (1-yr): | $\Delta P$, in. Hg | 0 | | 2.4 | 0.1 | 0 | |
| | Color | 1 | | 3 | 5 | 4 | |
| Drum 10 (1-yr): | $\Delta P$, in. Hg | 1.9 | | 0.1 | 3.5 | | |
| | Color | 1 | | 1 | 2 | | |
| Drum 14 (16-wk): | $\Delta P$, in. Hg | 0 | 0.5 | 0 | 7.6[a] | | |
| | Color | 2 | 1 | 5 | 6 | | |
| Drum 19 (16-wk): | $\Delta P$, in. Hg | 0 | | 30 | 6.9[a] | | |
| | Color | 2 | | 4 | 3 | | |
| Drum 20 (16-wk): | $\Delta P$, in. Hg | 0 | | 0.9 | 2.5 | 30[a] | 4.3 |
| | Color | 1 | | 2 | 2 | 4 | 2 |

NOTE: This table includes only those samples for which a definite breakpoint temperature was obtained with duplicate tests at the breakpoint, and for which the filter plugging at the breakpoint was significant.

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a. Pressure drop recorded before completion of full 240-minute test.

breakpoint 25°F downward from the value that would have been established by preheater rating.

From the results that have been discussed here and from an examination of the overall results (Table 4), it is concluded qualitatively that the breakpoint ratings are not influenced to more than 25°F by repeatability deviations. This conclusion applies only to a series of consecutive tests performed on a given fuel sample. Over the course of the program, long-term drifts in the test severity are considered quite probable. There is no way to detect such drifts, since no standard, stable reference fuel is available. Therefore, no quantitative measure is available for the probable deviations of the breakpoints from a hypothetical "true" value. Such deviations are almost surely greater than the 25°F indicated for a given test series. For purposes of further discussion, it will be assumed that breakpoints listed in this report are valid within a ±50°F range. This somewhat pessimistic assumption is consistent with the deviations found among the drums of fresh fuel. Such an assumption also explains the up-and-down minor variations in apparent thermal stability of the fuel during the storage period.

For the purposes of this report, the fuel coker results serve primarily to establish broad trends in the effects of different materials, but cannot be used to draw very fine comparisons.

One other item of data from Table 4 should be mentioned here   For the fuel coker tests in this program, samples were prefiltered through No.12 Whatman paper, as specified in the standard fuel coker test, ASTM D 1660-64. Prefiltration through a 0.45μ membrane filter is often used for samples to be run in the CRC research and modified cokers. The only tests performed in this program for a comparative check of the two prefiltration methods were two tests on 16-week fuel from Drum 20 (without material specimen) at a fuel coker preheater temperature of 650°F:

| Paper prefiltration: | Test filter $\Delta P$ 2.5 in., preheater 2(3) |
| Membrane prefiltration: | Test filter $\Delta P$ 0.9 in., preheater 2(3) |

Although these two test results "straddle" the filter breakpoint criterion of 2.0 in. Hg, the deviation is small; the results may be considered as excellent checks, certainly better than the deviations normally observed in this program between duplicate filter plugging results at the breakpoint (see Table 6). For this particular fuel sample, then, the choice of prefiltration method had no significant effect on breakpoint. For other fuel samples, particularly those giving severe filter plugging at the breakpoint, the prefiltration method could have a much more significant effect

Thermal stability breakpoints for all test series are summarized in Table 7. It will be observed that, for some of the fuel samples, breakpoints tended to fall off during the first part of the year's storage and then recover to a higher level during the later periods. Such a trend is suggested by the data for many of the fuel samples, but the only one for which the trend was unquestionably

2f

# TABLE 7. SUMMARY OF THERMAL STABILITY BREAKPOINTS

| Specimen | Breakpoint (°F) after 130°F storage for periods indicated | | | |
|---|---|---|---|---|
| | Fresh | 16 weeks | 35 weeks | 1 year |
| None (cold storage) | 625 | — | — | — |
| None (cold storage) | >675 | — | — | — |
| None | --- | 625 | 650 | 650 |
| None | >675 | 650 | 650 | >675 |
| None, wet | — | >675 | 625 | 550 |
| Steel | >675 | 625 | 600 | 675 |
| Rusted steel | >675 | 575 | 500 | 550 |
| Rusted steel, wet | >675 | >675 | 600 | 600 |
| Stainless steel | >675 | 675 | 650 | 575 |
| Aluminum (6061) | >675 | 675 | 625 | 600 |
| Aluminum (5052) | >675 | 675 | 675 | 600 |
| Brass | >675 | 575 | ≤575 | 525 |
| Brass, wet | 625 | ≤475 | — | 525 |
| Bronze | >675 | ≤475 | — | 550 |
| Hose liner "A" | 625 | ≤375 | — | ≤350 |
| Hose liner "B" | 650 | ≤400 | — | 400 |
| Coupling gaskets | >675 | 450 | — | ≤350 |
| Coating "M" | >675 | 650 | 625 | 675 |
| Coating "N" | >675 | 675 | 625 | 600 |
| Plug valve grease | >675 | >675 | 600 | 675 |
| F/S element | 650 | 625 | 600 | 625 |

27

significant was the sample stored with brass, which recovered more than 150°F in breakpoint during the last storage period. Similar recoveries may have been made by the samples stored with "brass, wet" and with bronze, but the data are not sufficient to define magnitude of the recovery.

Initial breakpoints on the fresh fuels ranged from 625°F to above 675°F. As mentioned previously, only broad trends can be described with assurance. For purposes of discussion, the following classification is used for degree of deterioration, based on breakpoint after storage:

A — no significant deterioration (625°F or higher)
B — slight deterioration       (575-600°F)
C — definite deterioration      (500-550°F)
D — severe deterioration        (475°F or lower)

Using the worst (lowest) breakpoint obtained on a given fuel sample during the storage period, the following ratings can be assigned:

| Material Specimen | Ratings | | |
|---|---|---|---|
| None | A | | |
| None, wet | | C | |
| Steel | B | | |
| Rusted steel | | C | |
| Rusted steel, wet | B | | |
| Stainless steel | B | | |
| Aluminum alloys | B | | |
| Copper-base alloys | | | D |
| Synthetic rubbers | | | D |
| Tank coatings | A B | | |
| Plug valve grease | B | | |
| F/S element | B | | |

Thus, the only materials that were extremely and consistently deleterious to fuel thermal stability were the copper-base alloys and the Buna N type rubbers. Both of these classes of materials caused severe drops in thermal stability even after 16 weeks of hot-room storage. The brass gave a relatively slow effect in comparison with that of the bronze, but the use of wet fuel with the brass accelerated the degradation.

Of the steels and aluminums, only the rusted steel stored in dry fuel gave any substantial degradation. None of the nonmetallic materials other than the rubbers gave any substantial degradation.

The effect of water on the degradation of thermal stability was different for the three cases tested. Without any material specimen, the water had a significant adverse effect on degradation. With rusty steel, the water was slightly beneficial. With brass, the water merely accelerated the initial stage of degradation.

28

B.    Metal Contents

The results of trace metal analyses performed by Monsanto Research Corporation are listed in Table 8. As indicated previously (Section III-C), the lower limits of detection for these metals are 5-10 parts per billion, with an estimated accuracy within 20%. The metal content data (Table 8) indicate that small but significant amounts of all four metals, copper, iron, lead, and zinc, appeared in the majority of the fuel samples that were tested. Metal contents on the order of 10 to 25 ppb are classed as significant in terms of the analysis itself, but are no doubt subject to metal pickup in sample transfer and handling. These samples were all taken in ordinary sample cans which had been cleaned with solvents but had not been subjected to any exhaustive cleaning procedures. Likewise, containers and utensils used in sampling and transfer were clean only in terms of normal fuel laboratory procedures. For these reasons, it is believed that metal contents on the order of 25 ppb should not be construed as representing significant pickup of metal from the drum or material specimen. Further, it will be noted from an inspection of the data (Table 8) that metal contents in that range bear no logical relation to the types of material specimens used.

Above 30 ppb, the picture is quite different: the metal contents in most cases can be related to material specimens present. Although there is obviously a "grey area" and not a sharp transition from nonsignificance to significance, it is useful for purposes of discussion to consider metal contents above 30 ppb as significant.

On this basis, it will be noted (Table 8) that there was no significant metal pickup in the initial fuels or in those stored in the hot room without any added material. Significant metal pickups and maximum values (ppb) recorded for the fuels stored in the hot room with material specimens and/or water are as follows:

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| No specimen, wet | Fe | 50 | Cu | 35 | | | |
| Steel | | | None | | | | |
| Rusted steel | Fe | 63 | | | | | |
| Rusted steel, wet | Fe | 135 | | | | | |
| Stainless steel | Fe | 42 | Cu | 54 | | | |
| Aluminum alloys | | | None | | | | |
| Brass | | | Cu | 273 | | Zn | 144 |
| Brass, wet | Fe | 87 | Cu | 458 | | Zn | 83 |
| Bronze | Fe | 47 | Cu | 274 | | | |
| Hose liner "A" | Fe | 104 | | | Pb 528 | Zn | 2700 |
| Hose liner "B" | | | | | Pb 232 | Zn | 4600 |
| Coupling gaskets | | | | | Pb 240 | Zn | 6900 |
| Coating "M" | Fe | 38 | | | | | |
| Coating "N" | Fe | 38 | | | | | |
| Plug valve grease | | Fe not determined; others—none | | | | | |
| F/S element | | Fe not determined; others—none | | | | | |

## TABLE 8.  METAL CONTENTS OF FUELS FROM HOT STORAGE

| Specimen | Weeks storage | Metal content, parts per billion | | | | Drum no. |
|---|---|---|---|---|---|---|
| | | Copper | Iron | Lead | Zinc | |
| None, cold storage | 0 | 6 | 15 | 10[a] | — | 1 |
| None, cold storage | 0 | 4 | 24 | 10[a] | — | 21 |
| None | 16 | — | 16 | — | — | 2 |
| | 35 | <5 | 8 | <10 | 18 | 2 |
| | 52 | 14 | 16 | 22 | 11 | 2 |
| None | 16 | <5 | 27 | 13 | 9 | 20 |
| | 52 | <5 | 15 | <10 | 10 | 20 |
| None, wet | 16 | — | 50 | — | — | 3 |
| | 35 | <5 | 52 | <10 | 9 | 3 |
| | 52 | 35 | 49 | <10 | 17 | 3 |
| Steel | 16 | — | 23 | — | — | 14 |
| | 16[b] | — | 13[b] | — | — | 14 |
| | 52 | 13 | 19 | <10 | 16 | 14 |
| | 52[b] | <5[b] | 18[b] | <10[b] | <5[b] | 14 |
| Rusted steel | 16 | — | 16 | — | — | 15 |
| | 16[b] | — | 24[b] | — | — | 15 |
| | 52 | 11 | 63 | 12 | 24 | 15 |
| | 52[b] | 12[b] | 33[b] | <10[b] | 12[b] | 15 |
| Rusted steel, wet | 16 | — | 50 | — | — | 16 |
| | 16[b] | — | 135[b,c] | — | — | 16 |
| | 52 | 26 | 91 | <10 | 15 | 16 |
| | 52[b] | 28[b] | 57[b] | <10[b] | 16[b] | 16 |
| Stainless steel | 16 | — | 56 | — | — | 4 |
| | 52 | 54 | 42 | <10 | 19 | 4 |
| Aluminum (6061) | 52 | 11 | 16 | <10 | 11 | 5 |
| Aluminum (5052) | 52 | <5 | 14 | <10 | 10 | 6 |

a.  Other drums sampled for analysis of lead content prior to hot storage and prior to introduction of specimen gave following results:

      Drum 8 - 14 ppb        Drum 10 - 22 ppb
      Drum 9 - 23 ppb        Drum 11 - 88 ppb

b.  Sample filtered through 0.8µ membrane filter prior to analysis.

c.  Average of four determinations:  96, 123, 165, and 157 ppb.

TABLE 8. METAL CONTENTS OF FUELS FROM HOT STORAGE (Cont'd)

| Specimen | Weeks storage | Metal content, parts per billion | | | | Drum no. |
| | | Copper | Iron | Lead | Zinc | |
|---|---|---|---|---|---|---|
| Brass | 16 | 269 | — | 15 | 144 | 7 |
| | 52 | 273 | 16 | <10 | 43 | 7 |
| Brass, wet | 16 | 458 | — | 18 | 77 | 8 |
| | 52 | 332 | 87 | 13 | 83 | 8 |
| Bronze | 16 | 261 | — | 23 | 14 | 9 |
| | 52 | 274 | 47 | <10 | 28 | 9 |
| Hose liner "A" | 16 | 17 | 29 | 52 | 2700 | 11 |
| | 52 | <5 | 104 | 33 | 2250 | 11 |
| Hose liner "B" | 16 | 9 | 11 | 232 | 4600 | 18 |
| | 52 | 14 | 25 | 34 | 1570 | 18 |
| Coupling gaskets | 16 | 19 | 5 | 240 | 6900 | 13 |
| | 52 | 24 | <5 | 98 | 5900 | 13 |
| Coating "M" | 52 | 5 | 38 | 14 | 14 | 17 |
| Coating "N" | 16 | <5 | <5 | <10 | 11 | 19 |
| | 52 | <5 | 38 | 21 | 17 | 19 |
| Plug valve grease | 52 | <5 | — | 11 | 7 | 10 |
| F/S element | 52 | 6 | — | 11 | 16 | 12 |

The pickup of iron in all of the "wet" drums is quite evident. As described in a later section, some coating damage and rusting were observed in these drums after completion of the storage period, so the pickup of iron is quite understandable. Iron was also picked up from the rusted steel under dry conditions, but not from the clean sandblasted steel. Iron also showed up in a number of the other samples, including several where it could hardly come from the material specimen. Because of the prevalence of iron in storage and sample containers, significant iron contents may show up more or less at random in any of the samples. Difficulties were encountered in repeatability of the iron content results on one sample (rusted steel, wet), even though this sample was filtered through a membrane filter prior to analysis.

The fuel samples stored with uncoated mild steel did not pick up significant amounts of any metal other than iron.

The fuel sample stored with Type 304 stainless steel picked up significant amounts of iron and copper. The copper pickup was entirely unexpected, considering the normally low copper contents of this alloy and its good corrosion resistance. After completion of the storage test, the stainless steel specimen was analyzed; the following results were obtained:

|        | Analysis | AISI Type 304 Specs |
|--------|----------|---------------------|
| Ni, %  | 8.69     | 8.0-12.0            |
| Cr, %  | 18.44    | 18.^-20.0           |
| Si, %  | 0.60     | 1.0 max             |
| Mn, %  | 1.03     | 2.0 max             |
| P, %   | 0.020    | 0.045 max           |
| S, %   | 0.014    | 0.030 max           |
| C, %   | 0.04     | 0.08 max            |
| Ti, %  | 0.01     | ---                 |
| Cu, %  | 0.19     | ---                 |
| Mo, %  | 0.18     | ---                 |
| Cb, %  | <0.01    | ---                 |

The analysis met all specifications, and the copper content is considered normal. Although the AISI specification does not have any limit on copper content, certain other specifications for Type 304 stainless steel (in forms other than sheet or strip) include a maximum copper content of 0.5%. With the specimen used in this test, significant pickup of copper by the fuel appears

32

very unlikely. By straightforward calculation, the copper content of 54 ppb in the final 15-gallon fuel sample is found to be equivalent to 2.38 mg of dissolved copper, in comparison with 1263 mg of copper in the original metal specimen. Leaching to this extent would imply removal of all of the copper from the outer 0.0001-in. layer of the metal specimen, which is virtually impossible in the absence of visible corrosion. The copper content of 54 ppb determined on the fuel could represent sample contamination during sampling and transfer, an erroneous analysis, or interference by some other element or elements.

No significant metal contents were found in any of the fuel samples stored with aluminum-alloy specimens. Nominal composition of the two alloys are:

    6061: Mg 1%, Si 0.6%, Cu 0.25%, Fe 0.7% max.
    5052: Mg 2.5%, Cr 0.25%

Aluminum alloys in the 5000 and 6000 series are commonly specified for fuel-system applications. Evidently the small amount of copper present in the 6000-series alloys does not have any tendency to dissolve in this fuel, based on the results presented here. Aluminum alloys with higher copper contents, such as the 2000 series, could well behave differently.

The fuel samples stored with brass or bronze all picked up relatively large amounts of copper, in the 260-460 ppb range. The highest of these were observed with brass present in wet fuel. Nominal compositions of the two alloys are:

    Free-cutting yellow brass, CA-360: Cu 61.5%, Pb 3.0%, Zn 35.5%
    Cast bearing bronze, SAE-660:      Cu 83%, Sn 7%, Pb 7%, Zn 3%

As might be expected, the fuels picked up significant amounts of zinc from the brass but not from the bronze. No significant pickup of lead was observed.

With the three synthetic rubber specimens, the fuels picked up large amounts of zinc and lead. Zinc oxide is used as a filler in these rubbers, and lead compounds are often used as additives. Zinc and lead contents of the fuels were considerably higher at 16 weeks than after one year (see Table 9).

The coated steel specimens, the plug valve grease, and the filter-separator element contributed no significant amounts of metals to the fuel except for minor amounts of iron with the coated steel.

33

C.    Condition of Specimens and Drums after Test

Although this program was not intended to investigate the effect of
fuel on materials, but rather the reverse, it was considered desirable to con-
duct at least a visual examination of all drums and specimens to detect any
gross changes.  After each drum had completed one year of storage and the
fuel sample had been removed, the drum was cut open for inspection of drum
interior and specimen.

One particular phenomenon noted with a fair number of the drums was
the appearance of a white, powdery deposit on the interior surface after the
fuel residues had evaporated.  Such deposits did not appear immediately, and
no doubt many instances went undetected.  Since this phenomenon was first
observed rather late in the program, and since it proved to be rather elusive,
no detailed records were kept.  No attempt was made to identify the deposits.
It appears probable that they represent a change in the surface of the coating
during the year's storage.

Of the two drums that were stored with dry fuel without any material
specimen, one drum (No. 2) was in excellent condition after test, with no
visible deterioration of the coating.  The other drum (No. 20) had numerous
pinholes in the epoxy coating, and the coating appeared much lighter in color
than the coatings in the other drums.  The poor condition of this coating is
attributed to improper application during manufacture of the drum, rather
than to any effect of the fuel on the coating.  Despite the poor condition of the
coating, no significant amount of iron was picked up by the fuel (see Table 8).

Of the three drums stored with wet fuel, two showed serious deteriora-
tion of the interior coating.  The drum with no material specimen showed
peeling of the coating at the bung and considerable rusting; the water layer
was dark brown, with rust and epoxy particles throughout the drum.  The
drum with wet fuel and rusty steel showed numerous areas of coating damage
(possibly mechanical damage) and rust pits in these areas.  The drum with
wet fuel and brass had no coating damage whatever.

Other than the coating breakdowns just discussed, the only other
instance of any coating damage occurred with the bronze specimen, where
the relatively heavy specimen apparently inflicted mechanical damage to the
coating during drum handling operations.

None of the metal specimens appeared to be changed in any way after
test, except that the rusted steel specimen in the wet-fuel drum acquired some
nodular corrosion products.

The rubber specimens did not undergo any change that could be
detected by casual examination, except that the coupling gaskets tended to
"smudge" rather badly after completion of the year's storage.  The coated

steel specimens appeared to be in excellent condition, except for some bare areas on the end of the "N"-coated specimen, attributed to mechanical damage. The plug valve grease was still present in the same general form as when it was put into the drum. The sectioned filter-separator element showed no visible change except for having acquired a brownish tint on the outer fabric "sock" material.

## D.    Discussion of Results

In reviewing the results of this program, it should be kept in mind that the storage tests were run with relatively large ratios of specimen surface to fuel. For the metal and coated specimens, this ratio was 2.55 $in^2$/gal; for the rubber specimens, the ratio was 5.8-13.2 $in^2$/gal. The amounts of air available to the fuel were relatively high, since each drum of fuel was aerated initially and at each sampling, and the air/fuel space ratio in the drums ranged from about 0.2 at the start of the storage period to about 2.7 at the end. The storage temperature of 130°F and the storage period of one year, along with the other conditions mentioned, add up to a severe set of conditions in comparison with what fuels will usually encounter in storage and handling systems.

These facts have been mentioned to emphasize the point that the results from this program cannot be used to assign any safe storage life for fuels of this type in systems containing the given materials. Instead, the results can be used to classify materials as safe or unsafe for use in systems handling fuels of high thermal stability. They do not answer the question "How unsafe?" Materials that are highly deleterious to thermal stability could possibly be tolerated in fuel systems of this nature if the surface areas involved or the fuel residence time in these particular areas of the system were sufficiently small.

The particular fuel used in this program, a 75-luminometer low-volatility naphthentic fuel, did not undergo any detectable change in thermal stability when stored for one year at 130°F in epoxy-lined drums. At most minor deterioration in thermal stability was observed with specimens of mild steel, Type 304 stainless steel, 5000- or 6000-series aluminum, MIL-C-4556B coating materials, plug valve grease, or one type of filter-separator element. Somewhat more deterioration was observed when rust was present in the drums. Very severe deterioration was observed when the drums contained specimens of brass, bronze, or Buna N type rubbers.

With the brass and bronze specimens, the deterioration in fuel thermal stability is related to the well-known effect of soluble copper, which was found in the fuels to the extent of 260-460 parts per billion. With the rubber specimens, the deterioration in fuel thermal stability may be related either to the effect of soluble lead and zinc, which were found in amounts up to several thousand parts per billion, or to the effect of organic materials such as

plasticizers that would be leached fro    the rubber during storage.  Plasticizer
leaching undoubtedly occurs, but ther·  ·  ·o way of establishing how much this
contributes to the loss in fuel thermal stability, apart from the effect of the
lead and zinc compounds.

Attempts to correlate fuel thermal stability with metal contents have
not yielded any valuable information beyond the broad trends already
described.

Although the effect of copper-base alloys on fuel thermal stability has
been well known for many years, the effect of synthetic rubbers has been less
publicized.  Of the three rubber specimens used in this program, two were
liner materials from fuel hose meeting an Air Force specification for low-
temperature hose.  Such liner materials are highly plasticized rubbers of
the general type of Buna N and are known to be highly subject to leaching of
plasticizer by fuel.  The other specimen, consisting of coupling gaskets, is
believed to be a less highly plasticized material, although no specific infor-
mation is available.  In view of the extremely deleterious effect of all three
rubber specimens on fuel thermal stability, it would have been of interest to
have included in the program other elastomers used in fuel handling particular
systems, in particular fluorinated rubbers and polyurethane elastomers.

Since copper-base alloys and Buna N type rubbers can be extremely
damaging to fuel thermal stability under some conditions, the ideal system
for handling thermally stable fuels would exclude such materials completely.
The practical difficulties in excluding these materials are well known.
Nevertheless, this would be the only safe way to guard against the effects of
these materials.  As an example, consider the use of refueling hose with
these types of fuels.  In terms of the total volume of fuel handled by a hose
in normal service, the possibility of any gross effects on thermal stability
are indeed very remote.  However, assuming that a new hose is put into
service on thermally stable fuel and then stands idle for a period of several
weeks, the residual fuel in the hose quite probably would be degraded very
seriously.  Then, the next refueling operation would move this fuel into an
aircraft tank or tanks.  Unless diluted very greatly with "fresh" fuel, the
residual fuel from the hose could be a source of trouble in the aircraft.  Some-
what the same considerations will apply to other elastomers and metals of the
fuel handling system.

Although this program has pointed out the possibility of thermal
stability degradation from copper-base alloys and Buna N type rubbers, it
has given very little information on tolerance levels in the system.  The only
comparative data relating to surface areas may be expressed in terms of
specimen area per gallon of original fuel sample:

| | | |
|---|---|---|
| Brass and bronze | 2.6 $in^2$/gal | Severe effects |
| Rubbers | 5.8-13.2 $in^2$/gal | Severe effects |
| Rubber in F/S element | 0.005 $in^2$/gal | Very little effect |

36

Thus, it can be seen that very small amounts of rubber are not likely to give much fuel deterioration, even under severe storage conditions.

Information of this type for design purposes could be obtained by "practical" type tests in either actual full-scale fuel systems or simulated systems. For example, in a refueler equipped with the normal complement of brass valves, bushings, and couplings and ordinary low-temperature fuel hose, a load of fuel could be held for several days or weeks with periodic recirculation in order to demonstrate whether any serious fuel degradation would occur. Possibly more useful would be a large number of static storage tests with copper-base alloy and rubber specimens, in which storage time, temperature and specimen-to-fuel ratio were varied systematically. Unfortunately, the relatively heavy demands of the fuel coker test in terms of fuel sample and running time will soon drive such a program beyond the bounds of practicality. It is suggested that, as a prelude to any such program, it would be useful to establish relationships between copper content and thermal stability for the particular fuel, as well as between zinc and/or lead contents and thermal stability if these metals are found to be controlling factors with the particular rubber involved. If such relationships can be established, they would be very useful in storage programs with small amounts of fuel, as well as in interpretation of field data.

# SECTION V

## CONCLUSIONS AND RECOMMENDATIONS

Copper-base alloys and Buna N type rubbers can be extremely harmful to the thermal stability of one particular type of high-quality special-purpose jet fuel. This fuel, a 75-luminometer, low-volatility naphthenic fuel, can be degraded from its initial thermal stability level of 625°F or higher down to a level below 400°F, i.e., to about the level of conventional JP-4. Such degradation is accompanied by the pickup of rather large amounts of copper from the copper-base alloys and of lead and zinc from the rubbers. Such severe degradation would not be expected to occur in normal operation of a fuel handling and storage system but could become a problem in intermittently operating equipment.

This particular fuel is stable for at least a year of storage at 130°F in lined drums with periodic aeration. Materials commonly used in fuel systems, other than copper-base alloys and Buna N type rubbers, do not cause serious degradation of the fuel under these conditions. However, combinations of water, steel, and rust can have deleterious effects on storage and thermal stability.

Based on the work reported herein, present criteria for ground fuel systems for handling special fuels are well founded in their exclusion of copper-base alloys and bare steel. The major materials of construction are aluminum, stainless steel, and coated steel. Within these classes, the materials used in ground fuel handling systems should have no serious effects on thermal stability. The results reported herein point out the need for a more serious consideration of the role of elastomers in fuel degradation, since ordinary fuel hose material appears to be at least as bad as copper-base alloys in this respect. It is reasonable to suppose that fluorinated rubbers will be less deleterious than the Buna N types, but no data on the former were obtained in this program.

It is recommended that information should be developed on the amounts of copper that can be tolerated in thermally stable fuels. Such information can then be used as a baseline for determining to what extent it is essential to eliminate copper-base alloys from the systems. Similar information on lead and zinc contents will be useful in application to the use of conventional fuel-resistant rubbers, provided it can be proved that these metals are the controlling factor in the degradation caused by such rubbers.

# DOCUMENT CONTROL DATA - R & D

*(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)*

| 1. ORIGINATING ACTIVITY (Corporate author) | 2a. REPORT SECURITY CLASSIFICATION |
|---|---|
| Southwest Research Institute<br>San Antonio, Texas | UNCLASSIFIED |
| | 2b. GROUP |

**3. REPORT TITLE**

JET FUEL STABILITY AND EFFECT OF FUEL-SYSTEM MATERIALS

**4. DESCRIPTIVE NOTES (Type of report and inclusive dates)**

Technical Report - 1 March 1966 through 30 November 1967

**5. AUTHOR(S) (First name, middle initial, last name)**

Johnston, Robert K.
Monita, Charles M.

| 6. REPORT DATE | 7a. TOTAL NO. OF PAGES | 7b. NO. OF REFS |
|---|---|---|
| February 1968 | 38 | -- |

| 8a. CONTRACT OR GRANT NO. | 9a. ORIGINATOR'S REPORT NUMBER(S) |
|---|---|
| AF 33(615)-2327 | RS-515 |
| b. PROJECT NO.<br>8169, 3048 | |
| c.<br>Task Nos. 816901, 304801 | 9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report) |
| d. | AFAPL-TR-68-20 |

**10. DISTRIBUTION STATEMENT**

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| 11. SUPPLEMENTARY NOTES | 12. SPONSORING MILITARY ACTIVITY |
|---|---|
| | Air Force Aero Propulsion Laboratory<br>Air Force Systems Command<br>Wright-Patterson AFB, Ohio |

**13. ABSTRACT**

A thermally stable, low volatility, naphthenic type jet fuel was stored in lined steel drums for one year at 150°F with various metallic and nonmetallic specimens representing ground fuel system materials. Fuel thermal stability was measured periodically by means of a gas-drive fuel coker. The fuel itself remained thermally stable during storage and was not degraded seriously by steels, aluminums, coated steels, plug valve grease, or a filter-separator element. Water or rusty steel, when present during storage, degraded the fuel thermal stability significantly. Brass, bronze, and butadiene-acrylonitrile rubbers of the type used for fuel hose gave the worst degradation of fuel thermal stability, accompanied by significant pickup of copper from the brass and bronze and of zinc and lead from the rubbers.

DD FORM 1473

| 14. KEY WORDS | LINK A | | LINK B | | LINK C | |
|---|---|---|---|---|---|---|
| | ROLE | WT | ROLE | WT | ROLE | WT |
| Jet fuel | | | | | | |
| Fuel systems | | | | | | |
| Fuel stability | | | | | | |
| Hydrocarbon fuels | | | | | | |